

ADMINISTRATIVE  
REPORT

**DEPARTMENT OF  
PUBLIC HEALTH AND HUMAN SERVICES  
ENVIRONMENTAL LABORATORY**



BRIAN SCHWEITZER  
GOVERNOR

ROBERT E. WYNIA, M.D.  
DIRECTOR

STATE OF MONTANA

PHONE: (406) 444-2642  
FAX: (406) 444-2617

1400 BROADWAY  
P.O. BOX 4369  
HELENA, MT 59604-4369

Jon Hager  
Energy Laboratories, Inc.  
3161 East Lyndale  
Helena MT 59601

December 6, 2005

**SDMS Document ID**



**1061802**

Dear Mr. Hager:

On April 1, 2004, representatives of the Department of Public Health and Human Services Environmental Laboratory conducted an on-site evaluation of your laboratory. The purpose of this evaluation was to determine your laboratory's compliance with the analytical requirements of the Safe Drinking Water Act, as specified in 40 CFR part 141, for certification to conduct compliance testing of Montana's Public Drinking Waters.

We found Energy Laboratories, Inc, Helena, to be well organized, clean, and to have sufficient space to conduct drinking water compliance analyses. Laboratory staff were knowledgeable and courteous during the on-site evaluation.

Attached is a report summarizing the findings of the April 1, 2004 on-site evaluation, prepared by Judy Halm, Lee Harbour and Gayle LaBlanc. Please submit a Plan of Correction in response to the findings of this report, according to the time frame listed in the report.

Energy Laboratory's term of licensure will remain in effect until 3/1/2008, under the following conditions:

1. Laboratory personnel correct the deficiencies noted in the enclosed report within the allotted time;
2. Energy Laboratory continues to pay annual fees as required by ARM 37.12.310;
3. The laboratory continues to use the methods approved under the National Primary Drinking Water Regulations, 40 CFR 141, for compliance drinking water samples;
4. Laboratory personnel perform acceptably on twice-a year Performance Evaluation samples for the above methods, and results of those PE samples are sent to the Environmental Laboratory;
5. Energy Laboratory wishes to retain certification.

The required documentation of corrective actions and deadlines for completing the corrective actions are described in the enclosed report. Any questions concerning this report should be addressed to Judy Halm (406) 444-2642.

We wish to thank you for your cooperation during this on-site evaluation.

Sincerely,

Judy Halm  
Laboratory Certification Officer  
DPHHS Environmental Laboratory

### **On-Site Evaluation Report**

This report contains the results of the on-site laboratory evaluation conducted for Energy Laboratory in Helena, Montana. The purpose of this evaluation was to determine your laboratory's compliance with the analytical requirements of the Safe Drinking Water Act, as specified in 40 CFR part 141, for certification to conduct compliance testing of Montana's Public Drinking Waters.

References used during this on-site and for this report are listed below:

1. EPA publication EPA 815-B-97-001, "Manual for the Certification of Laboratories Analyzing Drinking Water", 4<sup>th</sup> edition;
2. "Standard Methods for the Examination of Water And Wastewater," 18<sup>th</sup> edition, APHA, AWWA, WEF
3. The Administrative Rules of Montana (ARM), Rule 37.12 Subchapter 3, "Licensure of Laboratories Conducting Analyses of Public Water Supplies"
4. 40 CFR part 141 – National Primary Drinking Water Regulations;
5. 40 CFR part 142 – National Primary Drinking Water Regulations Implementation;
6. 40 CFR part 143 – National Secondary Drinking Water Regulations;

This report will identify areas of deficiency noted during the on-site visit, will cite references where information about this deficiency can be found, and will list the correction required. In certain instances, recommendations will be offered which may improve the ease or quality of the analysis. These are suggestions only, and are not requirements for certification.

#### **Laboratory Response to Audit Report**

Within 90 days of receiving this report, please submit a Plan of Correction which addresses each deficiency noted in this report. If you have already made corrections, forward documentation of the corrections. If you have any questions concerning any of the items on this report, please call the Environmental Laboratory at (406) 444-2642.

Certification staff will review your Plan of Correction and will either approve the plan or recommend changes if any need to be made. Within 90 days of the approval of your Plan of Correction, you must provide to the Environmental Laboratory proof that the Plan has been implemented. Include any documentation which demonstrates this implementation.

If you feel you need technical assistance in solving any of the deficiencies listed in this report, you may request assistance from staff of the DPHHS Environmental Laboratory. A fee of \$250 per day, including travel expenses (ARM 37.12.310) will be charged for technical assistance.

**On-Site Evaluation for Inorganic and Physical Parameters  
Energy Laboratories  
Helena, Montana  
Prepared by Judy Halm, Chemist**

**Certified Parameters:**

**Nitrate + Nitrite; Nitrate; Nitrite by Automated Cadmium Reduction  
Total Dissolved Solids @180° C  
Alkalinity  
pH  
Specific Conductance  
Turbidity**

**1. Nitrate + Nitrite, Nitrate and Nitrite by Automated Cadmium Reduction**

The laboratory is using EPA Method 353.2, an approved method for the analysis of nitrate + nitrite, nitrate and nitrite. The analytical method and SOP were available in the laboratory.

- a) A Linear Calibration Range for nitrate + nitrite was not available. The method requires that a linear calibration range be determined every 6 months.

**Requirement:** Determine the Linear Calibration Range once every 6 months, and retain documentation of each determination. Forward a copy of the Linear Calibration Range determination, including all raw data, to the State Environmental Laboratory.

- b) The Laboratory Reagent Blank must be less than the Method Detection Limit. If the LRB is higher than the MDL, contamination should be suspected and corrective actions must be taken before analysis continues.

**Requirement:** If the LRB is higher than the Method Detection Limit (not the reporting limit), determine the source of contamination and correct before continuing analyses.

**Reference:** EPA Method 353.2 "Determination of Nitrate-Nitrite Nitrogen by Automated Colorimetry"; Revision 2.0, August 1993 Sections 9.2.2 and 9.3.1.

**2. Total Dissolved Solids at 180° C**

The laboratory is using Standard Methods 2540 C, an approved method for the analysis of TDS. The analytical method and SOP were available to the analysts. The following problem was noted:

- a) The glass fiber filters used for analysis are not rinsed with reagent-grade water before being used. Standard Methods 2540 C.3.a requires that the filters be rinsed with three successive 20-mL volumes of reagent grade water prior to use.

**Requirement:** Rinse each filter with three successive 20-mL volumes of reagent grade water prior to use.

**3. Turbidity**

EPA Method 180.1, an approved method, is being used for the analysis of Turbidity in drinking water. The analytical method and SOP were available to the analysts. The following problem was noted:

- a) Method 180.1, Revision 2, August 1993 requires that an Initial Demonstration of Performance be completed for each analyst who will be running this method. A Linear Calibration Range (LCR) must be determined initially and verified every 6 months, or whenever a significant change in instrument response is observed or expected (Method 180.1, 9.2.2). The Linear Calibration Range study was not available at the time of the on-site audit.

**Requirement:** Determine the Linear Calibration Range for the method every 6 months and retain the documentation for review. Forward a copy of the Linear Calibration Range determination, including all raw data, to the State Environmental Laboratory.

- b) Secondary standards are certified against primary standards which are located in the Billings Energy Laboratory facility. Copies of the certification must be kept in the Helena laboratory.

#### 4. Specific Conductance

Standard Method 2510 B is used for the analysis of Specific Conductance in drinking water. This is an EPA approved method. No deficiencies were noted.

#### 5. pH by Electrometric Probe

Standard Method 4500 H<sup>+</sup> B is used for the analysis of pH in drinking water. This is an approved EPA method. No deficiencies were noted.

#### 6. Alkalinity

Standard Method 2320 B is used for the analysis of alkalinity in drinking water. This is an EPA-approved method.

**Requirement:** Add the calculation for carbonate alkalinity to the Standard Operating Procedure.

#### 7. Ortho phosphorous

EPA Method 365.1 is used for the analysis of ortho phosphorous in drinking water. This is an approved EPA method. The following deficiency was noted:

- a) EPA Method 365.1 (9.2.2) requires that a Linear Calibration Range be "determined initially and verified every 6 months, or whenever a significant change in instrument response is observed or expected." Linear Calibration Range documentation was not available.

**Requirement:** Determine the Linear Calibration Range for ortho phosphorous at least every 6 months, and retain the documentation for review. Forward a copy of the Linear Calibration Range determination, including all raw data, to the State Environmental Laboratory.

#### 8. Sulfate – Turbidimetric Method

Standard Method 4500-SO<sub>4</sub><sup>2-</sup> E is a recommended method for the analysis of sulfate in drinking water. The following deficiency was noted:

- a) SM 4500-SO<sub>4</sub><sup>2-</sup> E requires stirring of the sample plus buffer plus BaCl<sub>2</sub> for 60 seconds plus or minus 2 seconds at a constant speed. No set stir time was being used in the laboratory.

**Requirement:** Determine a set stir time to be used for each sample. Update the SOP to reflect this change.

- b) **Linear Calibration Range** – although a Linear Calibration Range is not required in Standard Methods, it is recommended that the LCR be calculated at least every 6 months. See EPA drinking water methods for inorganic parameters for instructions on determining the Linear Calibration Range.

## **9. Fluoride by Ion Selective Electrode**

Standard Method 4500-F- C is used for the analysis of Fluoride in drinking water. This is an EPA-approved method. The following deficiencies were noted:

- a) Standard Methods (SM 1020 B.4) recommends the analysis of reagent blanks a minimum of 5% of the sample load. Reagent blanks were not being analyzed by the laboratory.

**Requirement:** Analyze a laboratory reagent blank at the rate of at least 5% of the sample load, or not less than after the initial calibration, after each 20<sup>th</sup> sample, and at the end of an analytical batch.

- b) Standard Methods (SM 1020 B.2) recommends the use of known additions (spikes) to determine accuracy of analytical results. Spikes were not being analyzed by the laboratory.

**Requirement:** Determine an appropriate spike level for individual samples and spike at least 10% of the samples in each batch.

- c) **Linear Calibration Range** – although a Linear Calibration Range is not required in Standard Methods, it is recommended that the LCR be calculated at least every 6 months. See EPA drinking water methods for inorganic parameters for instructions on determining the Linear Calibration Range.

**Drinking Water Certification On-Site Evaluation  
4/1/2004**

**Energy Laboratories, Inc.  
3161 E. Lyndale Ave.  
P.O. Box 5688  
Helena, Montana 59604**

**Method EPA 200.7  
Determination of Metals and Trace Elements in Water and Wastes  
Drinking Water Contaminants  
By Inductively Coupled Plasma - Atomic Emission Spectroscopy**

**Instrument: Spectro Ciros Model FCE-12, Axial View**

**Analyst: Jon Hager  
Primary Inorganic Contaminants: Ba, Cd  
Unregulated Inorganic Contaminants: Ni  
Secondary Inorganic Contaminants: Ag, Zn  
Provisional: As until January, 2006 (MDL Required 0.01 ppm)  
Provisional: Al, B, Be, Cr, Cu, Fe, Mn, Mo**

**Analyst: Roger Pasch  
Provisional: Ag, Al, B, Ba, Be, Cd, Cr, Cu, Fe, Mn, Mo, Ni, V, Zn Ca, Mg, Na, K  
Provisional: As until January, 2006 (MDL Required 0.01 ppm)**

**1. Method Detection Limits (MDLs)**

Method Detection Limits were analyzed according to the procedure outlined in Method 200.7. If additional confirmation is desired, it is recommended (not required) to reanalyze the MDLs on two more nonconsecutive days, taking the average of the three MDL values for a more appropriate estimation. For analytes with an MDL < 0.01 ppm, the standard concentration used for the MDL determination needs to be lowered to 3-5 times the MDL for a more realistic determination.

**References:**

- A. EPA Methods for the Determination of Metals in Environmental Samples, Supplement 1  
EPA Publication 600/R-94/111, May 1994  
Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4  
Section 9.2.4
- B. EPA Manual for the Certification of Laboratories Analyzing Drinking Water  
Criteria and Procedures, Quality Assurance, Fourth Edition  
EPA Publication 815-B-97-001, March 1997  
Appendix H, Page H-4, Section 2.3 Initial Demonstration of Capability

**2. Calibration of the ICP**

Method 200.7 states the calibration line should consist of a minimum of a calibration blank and a high standard, using replicates to provide optimal distribution of calibration standards. The Certification Manual recommends the curve be composed of at least three concentrations

in the linear portion of the curve. The QA Manual states (Page 14) that for most instruments, a calibration curve is composed of three to five standards covering the working range. Appendix C, Table 1, Page 2, indicates the initial calibration for metals consists of two points, a blank and a standard. The Method QA/QC Parameters in Appendix C, Page 1 for Method 200.7 also indicates the instrument initial calibration is composed of one standard and a blank. The QA Manual in Appendix C for Method 200.7 needs to be modified to reflect the use of a multi-point calibration curve for drinking water parameters.

**References:**

- A. EPA Methods for the Determination of Metals in Environmental Samples, Supplement 1  
EPA Publication 600/R-94/111, May 1994  
Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4  
Sections: 4.4, 11.4.4
- B. EPA Manual for the Certification of Laboratories Analyzing Drinking Water  
Criteria and Procedures, Quality Assurance, Fourth Edition  
EPA Publication 815-B-97-001, March 1997  
Appendix H, Pages H-6 and H-7, Section 2.4

**3. Initial Calibration Blank, Continuing Calibration Blanks and POLs**

Analysis of the calibration blank should always be less than the analyte IDL, but greater than the lower 3-sigma control limit of the calibration blank. The parameters in Appendix C, Table 1 for the Method Blank and the Method QA/QC Parameters, Page 1, for the ICB and CCB for Method 200.7 needs to be modified to reflect drinking water criteria.

Calibration blanks that consistently run greater than the analyte MDL indicate the MDL may no longer be valid and should be redetermined.

For future reference, the new edition of the EPA Certification Manual will require that laboratories not report values that are less than the lowest calibration standard.

For compliance monitoring a laboratory must be able to report analytical results that are equal to or less than the Maximum Contaminant Level for drinking water parameters. The POLs for Be and Cd are set at 0.01 ppm, above the MCL for both analytes. The reporting limits for Be and Cd need to be set at 0.004 and 0.005 ppm respectively.

**Reference:**

- A. EPA Methods for the Determination of Metals in Environmental Samples, Supplement 1  
EPA Publication 600/R-94/111, May 1994  
Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4  
Section: 9.3.4
- B. EPA Manual for the Certification of Laboratories Analyzing Drinking Water  
Criteria and Procedures, Quality Assurance, Fourth Edition  
EPA Publication 815-B-97-001, March 1997  
Appendix H, Pages H-5, Section 2.3.2
- C. Code of Federal Regulations, Title 40  
Inorganic Chemical Sampling and Analytical Requirements

## Section 141.23, Pages 345-356

**4. Laboratory Fortified Blank (LFB)**

The laboratory does analyze at least one LFB with each batch of samples. An aliquot of the LRB is fortified with all analytes of interest at a concentration of 0.2 mg/L or at a level that falls at the midpoint of the calibration curve, and at the same concentration as the matrix spike. The recovery of the LFB must fall within the required control limits of 85-115%. The laboratory must use LFB analyses data to assess lab performance. When sufficient performance data becomes available (usually about 20-30 analyses), control limits can be developed from the mean percent recovery and the standard deviation of the mean percent recovery. The laboratory-generated control limits must be equal to or better than the required control limits of 85-115%. The LFB must be carried through the entire preparation scheme as the samples, including sample digestion when applicable. The LFB criteria in Appendix C of the QA Plan for Method 200.7 needs to be included for undigested drinking waters, and the LFB should also be included in the initial sequence after calibration prior to analyzing samples.

**References:**

- A. EPA Methods for the Determination of Metals in Environmental Samples, Supplement 1  
EPA Publication 600/R-94/111, May 1994  
Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4  
Sections: 3.9, 7.10.3, 9.3.2, and 9.3.3
- B. EPA Manual for the Certification of Laboratories Analyzing Drinking Water  
Criteria and Procedures, Quality Assurance, Fourth Edition  
EPA Publication 815-B-97-001, March 1997  
Appendix H, Page H-7, Section 2.5.1

**5. Laboratory Duplicates and Laboratory Fortified Matrix (LFM)**

Providing there is adequate volume of solution, the same sample should be duplicated and fortified with the same analyte concentration as used for the LFB. The laboratory does duplicate and spike a minimum of 10% of routine samples, including drinking water samples requiring digestion. When sufficient performance data is available, (usually 20-30 samples) control charts can be developed to establish the upper and lower control limits, which must be equal to or better than the required control limits of 70-130%. The QA Manual in Appendix C for Method 200.7, Page 1, sets the frequency for spikes at 1/20, and needs to be updated to reflect the drinking water criteria for instrument analytical spikes.

**Reference:**

- A. EPA Methods for the Determination of Metals in Environmental Samples, Supplement 1  
EPA Publication 600/R-94/111, May 1994  
Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4  
Sections: 9.4.1, 9.4.2, 9.4.3

**6. Stock Solutions for Calibration Standards Not Dated When Opened**

The ICP stock standards must be dated when received and initialed and dated when opened.



**Reference:**

- A. EPA Manual for the Certification of Laboratories Analyzing Drinking Water  
Criteria and Procedures, Quality Assurance, Fourth Edition  
EPA Publication 815-B-97-001, March 1997  
Appendix H, Pages H-3 and H-4, Section 2.1 and Page H-11, Section 3.6

**7. Performance Evaluation Samples**

For a laboratory to become approved and maintain certification for conducting analyses of drinking water parameters, the lab must participate in at least two proficiency tests annually. Raw data for the proficiency samples needs to be readily accessible, the proficiency samples must be prepared according to the instructions, and multiple analyses are not allowed unless there is a problem with a particular analyte that requires reanalysis.

The raw data for WS-81 was not available. The results for Be, Cr, Cu, Fe, Mn, and Mo for WS-87 were determined from solutions that were prepared at a level 10 times more concentrated than the instructions indicated. Jon Hager's results for RTC04-1 were in compliance except for Al and V. Jon will need to complete proficiency tests for Al, Be, Cr, Cu, Fe, Mn, and Mo to maintain his certification for these elements. The results submitted by Roger Pasch for RTC04-1 were in compliance for all elements given provisional certification. Roger will need to complete another proficiency sample to obtain full certification for Ag, Al, B, Ba, Be, Cd, Cr, Cu, Fe, Mn, Mo, Ni, V, Zn, Ca, Mg, Na, and K.

**Reference:**

- A. Administrative Rules of Montana 3/31/99, Laboratories, Subchapter 3, Licensure of Laboratories Conducting Analyses of Public Water Supplies, Rule 37.12.320 (2-b)

**On-Site Evaluation for Organic Parameters  
Energy Laboratories  
Helena, Montana  
Prepared by Judy Halm, Chemist**

**Method 542.2  
Measurement of Purgeable Organic Compounds in Water  
By Capillary Column Gas Chromatography/Mass Spectrometry  
Revision 4.1  
1995**

**Certified Parameters: Regulated, Monitored and Unregulated VOCs**

---

No deficiencies were noted at this time.

**On-Site Evaluation for Microbial Parameters  
Energy Laboratories  
Helena, Montana  
Prepared by Lee Harbour, Chemist  
April 1, 2004**

Numbers refer to EPA Manual for the Certification of Laboratories Analyzing Drinking Water, Chapter 5.

Energy Labs in Helena is currently using Colilert for Drinking Water and are performing Fecal analysis using m-FC media and HPC analysis using Simplate.

As a general comment: Anything that the Helena Lab obtains or borrows from the Billings Lab needs to have all of the related QC paperwork with it.

- 3.2 Reference weights for balance are calibrated once every five years. Need to double check to see how often they need to be calibrated for chemistry.
- 3.3 Thermometer for 44.5° C water bath is off by 10° C from Reference thermometer. The thermometers need to be replaced if they are off by more than 1° C from the reference thermometer.
- 3.3 Certificates of Calibration are needed from the Billings lab for Reference thermometers.
- 3.5 Sterilization time and Total cycle time need to be recorded on autoclave log for each batch.
- 3.5 The automatic timing mechanism on autoclave needs to be checked quarterly with a stopwatch or other accurate timepiece. A 12-15 minute cycle should be complete within 45 minutes. This information needs to be recorded in the log as well.
- 3.5 Currently there is no way to calibrate the temperature on the autoclave short of sending it to the manufacturer. Therefore, a maximum-temperature-registering thermometer needs to be obtained and should be used with each batch or at least weekly. This information also needs to be recorded in the autoclave log.
- 3.5 Spore Strips or ampules need to be used monthly in addition to the heat indicating tape to prove sterility of items autoclaved and the results recorded in the autoclave log.
- 3.11 The membrane filters used in Helena were obtained from the Billings Lab. There should have been a use test performed on these samples. The results of the use test and any other QC information should be obtained and a copy kept in the Helena Lab as long as the items are in use.
- 3.13 Any disposable sterile pipets need to be checked for sterility once per lot. This information needs to be recorded.
- 4.3 Reagent Grade Water should be tested for TKN and TOC at least annually, if not monthly.
- 4.3 The BacT Quality of Water Test may be discontinued if the requirements for Type I and II water can be obtained according to the 18<sup>th</sup> Ed. of Standard Methods, Section 1080.